

PERFORMANCE DEMONSTRATION PROGRAM PLAN FOR RCRA CONSTITUENT ANALYSIS OF SOLIDIFIED WASTES

**Revision 8
December 2010**



**U.S. Department of Energy
Carlsbad Field Office
Office of the National TRU Program**

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Performance Demonstration Program Plan for RCRA Constituent Analysis of Solidified Wastes

**DOE/CBFO-95-1077
Revision 8**

December 2010

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TABLE OF CONTENTS

ACRONYMS	6
Significant Changes to RCRA Constituent PDP Plan, Revision 8	7
1.0 INTRODUCTION.....	8
1.1 General	8
1.2 Purpose.....	8
1.3 Scope and Frequency	9
2.0 PROGRAM COORDINATION	14
2.1 General Responsibilities	14
2.2 Program Assessment	16
2.3 Procurement	16
2.4 Training	16
3.0 PREPARATION AND DISTRIBUTION OF PDP SAMPLES.....	17
4.0 ANALYTICAL AND DATA REPORTING REQUIREMENTS	19
4.1 Sample Receipt and Chain-of-Custody	19
4.2 Analysis.....	19
4.3 Reporting.....	20
4.4 Analytical Records	22
5.0 EVALUATION OF PERFORMANCE DATA	23
5.1 Performance on Blanks	23
5.2 Detection and Accuracy	24
5.3 Precision of Duplicates	26
5.4 Precision of Replicates.....	26
5.5 Tentatively Identified Compounds (TICs).....	27
5.6 Overall Performance	28
6.0 REPORTING OF PERFORMANCE DATA	32
6.1 Summary of Data	32
6.2 Distribution of Reports	32
6.3 Laboratory Status	32
6.4 Quality Assurance Records	33
7.0 GLOSSARY.....	34
8.0 REFERENCES.....	37
APPENDIX A Example Chain-of-Custody Form.....	38
APPENDIX B Example Data Reporting Forms	40

LIST OF TABLES

<u>TABLE NO.</u>	<u>TITLE</u>	<u>PAGE</u>
Table 1.	Volatile Organic Compounds Target Analyte List	10
Table 2.	Semivolatile Organic Compound Target Analyte List	12
Table 3.	Metals Target Analyte List	13

LIST OF FIGURES

<u>FIGURE NO.</u>	<u>TITLE</u>	<u>PAGE</u>
Figure 1.	Organization and Information Flow for the RCRA PDP	15

ACRONYMS

CAS	Chemical Abstracts Service
CBFO	Carlsbad Field Office
COC	chain-of-custody
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
GC/MS	gas chromatography/mass spectrometry
HSG	headspace gas
IDL	instrument detection limit
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
NDA	nondestructive assay
PDP	Performance Demonstration Program
PRDL	program required detection limit
PRQL	program required quantitation limit
QA	quality assurance
QAO	quality assurance objective
QAPD	Quality Assurance Program Document
QC	quality control
%R	percent recovery
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
%RSD	percent relative standard deviation
SPC	sample preparation contractor
SVOC	semivolatile organic compound
SW-846	EPA (1998) Test Methods for Evaluating Solid Waste
TIC	tentatively identified compound
TRU	transuranic
VOC	volatile organic compound
VTSR	validated time of sample receipt
WAP	Waste Analysis Plan
WIPP	Waste Isolation Pilot Plant

Significant Changes to RCRA Constituent PDP Plan, Revision 8

General

1. The document was updated to reflect changes in the Hazardous Waste Facility Permit.
2. Editorial changes were incorporated as appropriate to improve technical clarity.

1.0 INTRODUCTION

1.1 General

The Performance Demonstration Program (PDP) for Resource Conservation and Recovery Act (RCRA) constituents distributes test samples for analysis of volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and metals in solid matrices. Each distribution of test samples is termed a PDP cycle. These evaluation cycles provide an objective measure of the reliability of measurements performed for transuranic (TRU) waste characterization.

The primary documents governing the conduct of the PDP are the *Quality Assurance Program Document* (QAPD; DOE/CBFO-94-1012) and the Waste Isolation Pilot Plant (WIPP) *Waste Analysis Plan* (WAP) contained in the Hazardous Waste Facility Permit (NM4890139088-TSDF) issued by the New Mexico Environment Department. The WAP requires participation in the PDP; the PDP must comply with the QAPD and the WAP. This plan implements the general requirements of the QAPD and the applicable requirements of the WAP for the RCRA PDP.

Participating laboratories demonstrate acceptable performance by successfully analyzing single-blind performance evaluation samples (subsequently referred to as PDP samples) according to the criteria established in this plan. PDP samples are used as an independent means to assess laboratory performance regarding compliance with the WAP quality assurance objectives (QAOs). The concentrations of analytes in the PDP samples address levels of regulatory concern and encompass the range of concentrations anticipated in waste characterization samples. The WIPP requires analyses of homogeneous solid wastes to demonstrate compliance with regulatory requirements. These analyses must be performed by laboratories that demonstrate acceptable performance in this PDP. These analyses are referred to as WIPP analyses, and the samples on which they are performed are referred to as WIPP samples. Participating laboratories must analyze PDP samples using the same procedures used for WIPP samples.

1.2 Purpose

The purpose of the RCRA constituent PDP is to demonstrate the capability of each participating laboratory, including mobile system vendors, to meet the data quality objectives stated in the WAP for the analysis of solidified waste samples. The U.S. Department of Energy (DOE), Carlsbad Field Office (CBFO) uses the PDP as one part of the assessment and approval process for the laboratories supplying services for the characterization of WIPP TRU waste. The process also includes the evaluation of method performance data submitted by the participating laboratory and the performance of quality assurance (QA) audits.

This PDP plan describes the detailed elements that the program comprises, including the nature of the test materials and the analyses required. This plan also identifies the criteria used for the evaluation of laboratory performance and the responsibilities of the RCRA PDP coordinator, the sample preparation contractor (SPC), and the participating laboratories. The CBFO is responsible for ensuring the implementation of this plan by designating the program coordinator and by providing technical oversight and coordination for the program. The program coordinator

will designate the PDP manager, who will coordinate the three PDPs (headspace gas [HSG], RCRA, and nondestructive assay [NDA] analyses). In turn, the PDP manager will assign a RCRA PDP coordinator who administers and coordinates RCRA PDP functions such as PDP sample component preparation, subcontractor oversight, scheduling, scoring, and generating summary reports. In addition to this PDP, there are two other PDPs that are described in three additional PDP plans: *Performance Demonstration Program Plan for Nondestructive Assay of Drummed Wastes for the TRU Waste Characterization Program* (DOE/CBFO-01-1005); *Performance Demonstration Program Plan for Nondestructive Assay of Boxed Wastes for the TRU Waste Characterization Program* (DOE/CBFO-01-1006); and *Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases for the TRU Waste Characterization Program* (DOE/CBFO 95-1076).

1.3 Scope and Frequency

In accordance with the WAP, all laboratories that perform RCRA constituent analyses of TRU wastes for disposal at WIPP are required to participate in this PDP. Satisfactory performance of PDP analyses is only one of several necessary conditions for certification to ship TRU waste to WIPP. The criteria for acceptable performance in the RCRA PDP are given in section 5 of this plan.

Acceptable performance in this PDP must be demonstrated initially by all participating laboratories prior to the analysis of WIPP samples. Subsequently, all participating laboratories shall be evaluated annually, as specified in the WAP. The primary cycle for PDP participation will therefore be annual (i.e., every twelve months, with a one-month grace period). In addition to the primary test cycle, the RCRA PDP coordinator may schedule a supplemental cycle prior to the next annual cycle. A supplemental cycle may be scheduled to qualify new laboratories, to requalify a laboratory that did not pass in the primary cycle, or to meet other programmatic needs at the discretion of CBFO. The participating laboratory(ies) may be required to provide funding for the supplemental cycle.

The PDP samples must be analyzed using the same analytical methods, procedures, and conditions of radioactivity confinement that the laboratory uses for the analysis of WIPP samples. These methods must have been developed and approved within the guidelines established by the WAP. Only the methods used in the PDP are acceptable to support the analysis of WIPP samples. The data generated during performance demonstration indicate the appropriateness of the method used as well as the performance of the laboratory.

The RCRA PDP consists of three components. These include analysis of VOCs, SVOCs, and metals in a solid matrix. The VOCs are further subdivided based on analytical methodology, resulting in four analyte categories: aqueous-extractable VOCs, purgeable VOCs, SVOCs, and metals. Target analytes are those required to establish hazardous waste determinations or to support determinations made using acceptable knowledge. The specific target analytes are listed in the WAP and the QAOs are listed in the Hazardous Waste Facility Permit, Attachment C3. Target analytes for VOCs, SVOCs, and metals and their associated QAOs are listed in tables 1, 2, and 3, respectively. Laboratories are required to participate in the program components corresponding to the analyses that they will be using for WIPP waste characterization.

Table 1. Volatile Organic Compounds Target Analyte List

Analyte	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL (mg/kg)	PRQL (mg/kg)
<i>Purgeable VOCs</i>					
Acetone ^f	67-64-1	≤50	60–150	10 ^e	100
Benzene	71-43-2	≤45	37–151	1	10
Bromoform	75-25-2	≤47	45–169	1	10
Carbon disulfide	75-15-0	≤50	60–150	1	10
Carbon tetrachloride	56-23-5	≤30	70–140	1	10
Chlorobenzene	108-90-7	≤38	37–160	1	10
Chloroform	67-66-3	≤44	51–138	1	10
1,4-Dichlorobenzene ^b	106-46-7	≤60	18–190	1	10
1,2-Dichlorobenzene ^b	95-50-1	≤60	18–190	1	10
1,2-Dichloroethane	107-06-2	≤42	49–155	1	10
1,1-Dichloroethene	75-35-4	≤250	D-234 ^c	1	10
<i>trans</i> -1,2-Dichloroethylene	156-60-5	≤50	60–150	1	10
Ethyl benzene	100-41-4	≤43	37–162	1	10
Ethyl ether ^f	60-29-7	≤50	60–150	10 ^e	100
Methylene chloride	75-09-2	≤50	D-221 ^c	1	10
Methyl ethyl ketone ^f	78-93-3	≤50	60–150	10 ^e	100
1,1,2,2-Tetrachloroethane	79-34-5	≤55	46–157	1	10
Tetrachloroethylene	127-18-4	≤29	64–148	1	10
Toluene	108-88-3	≤29	47–150	1	10
1,1,1-Trichloroethane	71-55-6	≤33	52–162	1	10
1,1,2-Trichloroethane	79-00-5	≤38	52–150	1	10
Trichloroethylene	79-01-6	≤36	71–157	1	10
Trichlorofluoromethane	75-69-4	≤110	17–181	1	10
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤50	60–150	1	10
Vinyl chloride	75-01-4	≤200	D-251 ^c	1	4
<i>meta</i> -Xylene ^d	108-38-3	≤50	60–150	1	10
<i>para</i> -Xylene ^d	106-42-3	≤50	60–150	1	10
<i>ortho</i> -Xylene	95-47-6	≤50	60–150	1	10

CAS, Chemical Abstracts Service; %RSD, percent relative standard deviation; RPD, relative percent difference; %R, percent recovery; MDL, method detection limit (maximum permissible value); PRQL, program required quantitation limit calculated from the toxicity characteristic level for benzene assuming a 25 g sample, 0.5 L of extraction fluid, and 100 percent analyte extraction.

- | | |
|---|---|
| a. Criteria apply to PRQL concentrations. | e. An estimate, to be determined. |
| b. Can also be analyzed as an SVOC. If analyzed as an SVOC, the QAOs listed in table 2 then apply. | f. Can be analyzed as either an aqueous extractable or as a purgeable VOC. |
| c. Detected; result must be greater than zero. | g. Required only for homogenous solids and soil/gravel waste from the Savannah River Site. |
| d. <i>meta</i> - and <i>para</i> -xylene are not separable by typical analytical methods and are usually reported as the sum of the two isomers. In this case, the combined MDL and PRQL remain 1 and 10 mg/kg, respectively. | h. Required only for homogeneous solids and soil/gravel from Oak Ridge National Laboratory and the Savannah River Site. |

Table 1. Volatile Organic Compounds Target Analyte List (cont.)

Analyte	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL (mg/kg)	PRQL (mg/kg)
<i>Aqueous-extractable VOCs</i>					
Acetone ^f	67-64-1	≤50	60–150	10 ^c	100
Butanol	71-36-3	≤50	60–150	10 ^c	100
Ethyl ether ^f	60-29-7	≤50	60–150	10 ^c	100
Formaldehyde ^g	50-00-0	≤50	60–150	10 ^c	100
Hydrazine ^h	302-01-2	≤50	60–150	10 ^c	100
Isobutanol	78-83-1	≤50	60–150	10 ^c	100
Methanol	67-56-1	≤50	60–150	10 ^c	100
Methyl ethyl ketone ^f	78-93-3	≤50	60–150	10 ^c	100
Pyridine ^b	110-86-1	≤50	60–150	10 ^c	100
CAS, Chemical Abstracts Service; %RSD, percent relative standard deviation; RPD, relative percent difference; %R, percent recovery; MDL, method detection limit (maximum permissible value); PRQL, program required quantitation limit calculated from the toxicity characteristic level for benzene assuming a 25 g sample, 0.5 L of extraction fluid, and 100 percent analyte extraction.					
a. Criteria apply to PRQL concentrations.			e. An estimate, to be determined.		
b. Can also be analyzed as an SVOC. If analyzed as an SVOC, the QAOs listed in table 2 then apply.			f. Can be analyzed as either an aqueous extractable or as a purgeable VOC.		
c. Detected; result must be greater than zero.			g. Required only for homogenous solids and soil/gravel waste from the Savannah River Site.		
d. <i>meta</i> - and <i>para</i> -xylene are not separable by typical analytical methods and are usually reported as the sum of the two isomers. In this case, the combined MDL and PRQL remain 1 and 10 mg/kg, respectively.			h. Required only for homogeneous solids and soil/gravel from Oak Ridge National Laboratory and the Savannah River Site.		

Table 2. Semivolatile Organic Compound Target Analyte List

Analyte	CAS Number	Precision ^a (%RSD or RPD)	Accuracy ^a (%R)	MDL (mg/kg)	PRQL (mg/kg)
<i>SVOCs</i>					
Cresols	1319-77-3	≤50	25–115	5	40
1,4-Dichlorobenzene ^b	106-46-7	≤86	20–124	5	40
1,2-Dichlorobenzene ^b	95-50-1	≤64	32–129	5	40
2,4-Dinitrophenol	51-28-5	≤119	D–172 ^c	5	40
2,4-Dinitrotoluene	121-14-2	≤46	39–139	0.3	2.6
Hexachlorobenzene	118-74-1	≤319	D–152 ^c	0.3	2.6
Hexachloroethane	67-72-1	≤44	40–113	5	40
Nitrobenzene	98-95-3	≤72	35–180	5	40
Pentachlorophenol	87-86-5	≤128	14–176	5	40
Pyridine ^b	110-86-1	≤50	25–115	5	40

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

MDL = Method detection limit (maximum permissible value)

PRQL = Program required quantitation limit calculated from the toxicity characteristic level for nitrobenzene assuming a 100 g sample, 2 L of extraction fluid, and 100 percent analyte extraction.

a. Criteria apply to PRQL concentrations.

b. Can also be analyzed as a VOC.

c. Detected; result must be greater than zero.

Table 3. Metals Target Analyte List

Analyte	CAS Number	Precision^a (%RSD or RPD)	Accuracy (%R)	PRDL^b (mg/kg)	PRQL (mg/kg)
Antimony	7440-36-0	≤30	80–120	10	100
Arsenic	7740-38-2	≤30	80–120	10	100
Barium	7440-39-3	≤30	80–120	200	2,000
Beryllium	7440-41-7	≤30	80–120	10	100
Cadmium	7440-43-9	≤30	80–120	2.0	20
Chromium	7440-47-3	≤30	80–120	10	100
Lead	7439-92-1	≤30	80–120	10	100
Mercury	7439-97-6	≤30	80–120	0.40	4.0
Nickel	7440-02-0	≤30	80–120	10	100
Selenium	7782-49-2	≤30	80–120	2.0	20
Silver	7440-22-4	≤30	80–120	10	100
Thallium	7440-28-0	≤30	80–120	10	100
Vanadium	7440-62-2	≤30	80–120	10	100
Zinc	7440-66-6	≤30	80–120	10	100

%RSD = Percent relative standard deviation

RPD = Relative percent difference

%R = Percent recovery

PRDL = Program required detection limit (i.e., maximum permissible value for instrument detection limit [IDL])

PRQL = Program required quantitation limit (mg/kg, wet weight basis)

^a≤30 percent precision applies when sample concentrations (certified values or certified + spiked values) are at least equal to the PRQL. If sample concentrations are less than the PRQL, the absolute difference between the two values shall be less than or equal to the PRDL.

^bPRDLs have been converted from the post-digestion aqueous concentrations specified in the WAP to the equivalent concentration in a 100 percent solid sample (i.e., the matrix used for RCRA PDP samples).

2.0 PROGRAM COORDINATION

2.1 General Responsibilities

The CBFO is the reviewing and approving authority for the RCRA PDP. Programmatic direction and oversight of the PDP are performed by the Office of the National TRU Program, which manages the PDP on behalf of the CBFO. Figure 1 summarizes the organizational flow of the RCRA PDP.

The PDP is performed periodically as described in the WAP and this document. A CBFO-designated organization functions as the PDP coordinator and technical advisor to CBFO. The program coordinator shall meet all of the responsibilities assigned by this plan in accordance with the requirements of the CBFO *Performance Demonstration Program Management Plan* (DOE/CBFO-01-3107). For the RCRA PDP, the RCRA PDP coordinator is responsible for the following:

- Ensures preparation, control, verification, and distribution of PDP standards by the SPC
- Distributes PDP cycle schedules to measurement-facility participants
- Confirms the scheduling of a PDP cycle at least two weeks before the planned start date
- Reviews procedures for PDP sample preparation
- Receives, reviews, and scores the analytical data
- Reports performance data as specified in this document
- Ensures that the records of participation and results of all PDP cycles are maintained in a retrievable condition
- Reviews changes in the QAPD or WAP that affect the PDP or this plan; revises this plan when necessary

The RCRA PDP coordinator provides independent technical oversight and coordination of the demonstration program to qualify participating measurement facilities and maintains a current list of the laboratories participating in the RCRA PDP. The CBFO must grant approval for each facility to be a participant in this PDP. Laboratories that are not current participants may petition the CBFO to be permitted to participate in the PDP.

Each participating laboratory is required to provide the RCRA PDP coordinator with the name, telephone number, fax number, and address of the contact persons responsible for administrative communications for the PDP. Each participating laboratory is also required to provide an address suitable for express package delivery of the PDP samples.

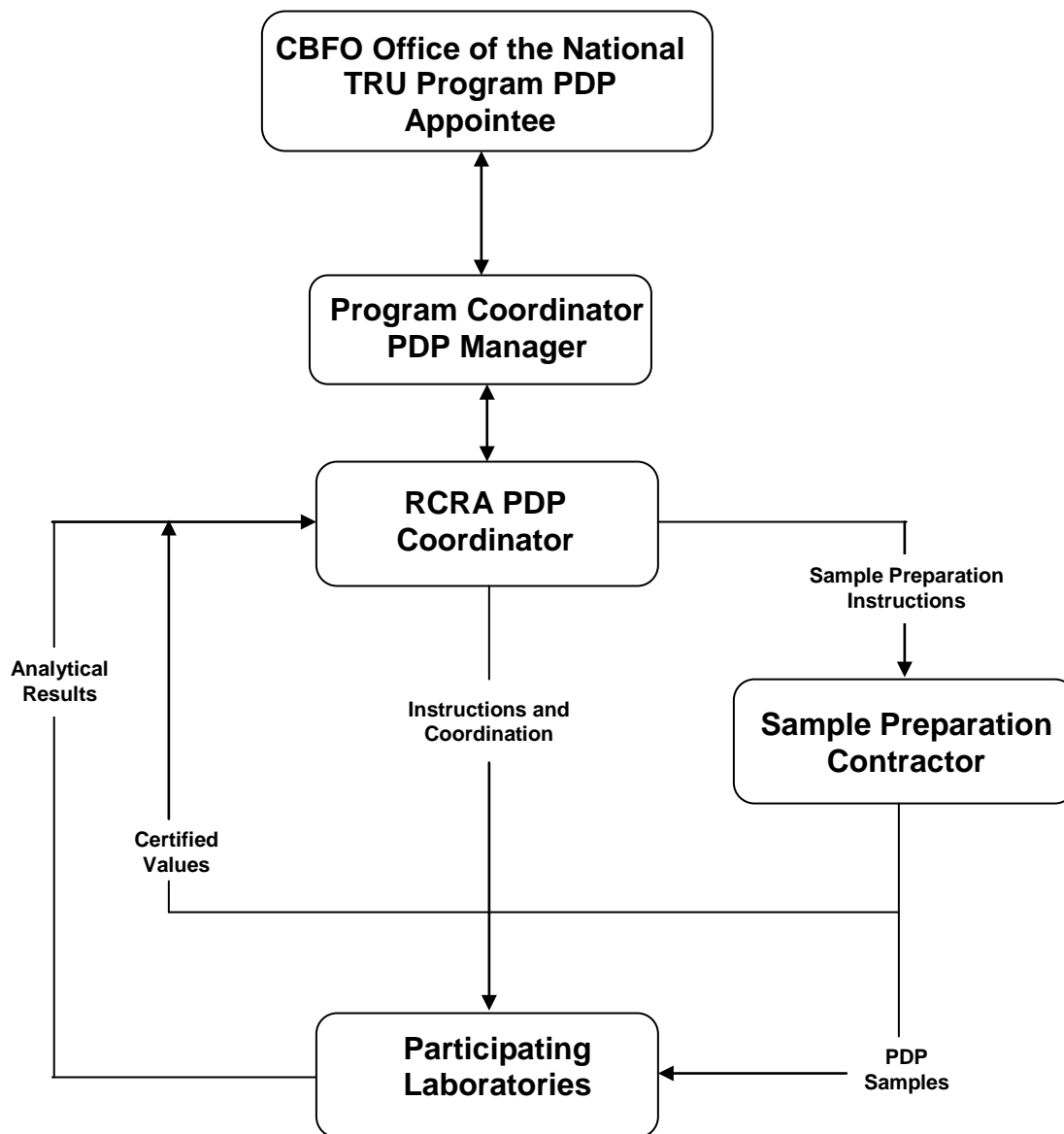


Figure 1. Organization and Information Flow for the RCRA PDP

The responsibilities of the SPC include:

- Preparing the PDP samples according to the specifications provided by the RCRA PDP coordinator
- Verifying that the concentrations of all target analytes in the PDP samples are within the specifications
- Shipping the PDP samples with all necessary identification, instructions, and custody forms. Result templates, when provided to the SPC by the RCRA PDP coordinator, may be distributed with the PDP samples
- Delivering documentation showing that the requirements of each PDP cycle have been met, including work plans, QA plans, analysis instructions, PDP sample identification keys, certificates of analysis, and verification data

2.2 Program Assessment

The PDP is routinely assessed for efficacy and appropriateness through several interrelated activities. These activities include CBFO's review and acceptance of the final testing results for each PDP cycle, as well as their review and approval of this plan. To assess the ongoing effectiveness of the PDP, the CBFO also considers reports and observations of the program coordinator, PDP manager, and RCRA PDP coordinator; feedback from program participants; and comments from other parties such as independent QA assessors. Such communications may take any documented form, including, but not limited to, routine program correspondence, meeting minutes, action items, formal review of program documents, assessment reports, and corrective action requests.

2.3 Procurement

Procurement activities necessary for conducting the PDP must comply with the QAPD. In accordance with the QAPD, the responsible purchasing organization maintains all procurement documents and performs all procurement activities.

2.4 Training

Each organization involved in conducting the PDP shall meet the training requirements of the QAPD. Organizations shall retain on file evidence that 1) personnel have the necessary program documents (controlled or uncontrolled, as applicable) for their use, and 2) personnel have read and understand program-governing documents pertinent to their duties in supporting the PDP. At a minimum, these documents include the WAP, the QAPD, and this plan.

3.0 PREPARATION AND DISTRIBUTION OF PDP SAMPLES

The PDP samples are prepared to cover the analytes of concern to the program and their range of expected concentrations. These analytes and associated QAOs are listed in tables 1, 2, and 3. Individual analytes may be present at concentrations ranging from approximately two times the PRDL/MDL to many times the PRQL. Appropriate blanks for each component will also be prepared. Final analyte concentrations in the PDP samples are left to the discretion of the RCRA PDP coordinator. Individual samples will not be limited to any specific number of analytes nor to a specific range of concentrations.

Individual samples intended for VOC or SVOC analyses may contain analytes not explicitly listed on the target analyte list. Laboratories are required to correctly identify and quantitate such analytes as tentatively identified compounds (TICs). In addition, any individual sample may contain potential interferents of interest to the program. In that event, the RCRA PDP coordinator will inform the laboratories of the presence and identity of any interferents. Interferents need not be explicitly reported. Participating laboratories are expected to be able to compensate for these interferents in performing the required analyses for target analytes.

The RCRA PDP coordinator shall arrange for one or more contractors to prepare the PDP samples. The SPC will have final responsibility for production of the PDP samples. The coordinator provides details on the required number and type of PDP samples that shall be prepared as well as the proposed spiking levels for each PDP sample.

The SPC prepares the PDP samples and provides verification analysis of the product. The SPC itself, or its subcontractor, must be able to verify/certify all target analyte concentrations in each of the PDP sample batches to meet the requirements described by the RCRA PDP coordinator. The SPC uses National Institute of Standards and Technology traceable target-analyte source materials (where available) directly, or through gravimetric means, to prepare the spiking solutions. The certificate of analysis shall provide proof of this traceability. Solid matrix source materials (i.e., soil or sand) shall be used for all analyte categories.

The SPC shall ship PDP samples with a delivery/chain-of-custody (COC) record (hereafter called the COC form). Appendix A contains an example COC form. The SPC shall identify each PDP sample according to the scheme provided by the RCRA PDP coordinator. Each PDP sample shall be labeled with a unique six-character identifier. The identifier allows the participant to determine the type of analysis required but not to associate it with any other samples in the program other than multiple aliquots of itself. The sample label shall also indicate the program (i.e., WIPP RCRA PDP), the cycle number, the container contents, the amount in grams, and any quality control (QC) instructions (e.g., use as matrix spike [MS] and matrix spike duplicate [MSD]).

The SPC shall forward analysis instructions to the participants along with the PDP samples. These instructions shall provide adequate detail to ensure that the laboratory appropriately prepares the samples for analysis (e.g., performs appropriate dilutions or consumes the entire contents of the vial). The RCRA PDP coordinator shall approve these analysis instructions before the samples are shipped.

After all PDP samples are shipped and verification analyses completed, the SPC shall deliver documentation showing that the requirements of the PDP have been met, including at a minimum:

- A copy of the analysis instructions for the participants
- A certificate of analysis for each batch of PDP samples
- Verification data, including a summary of results from the verification analysis for each PDP sample set, and results of all QC data generated related to the preparation and verification analysis of the PDP samples
- A table correlating the PDP sample identifiers, the certified analytical data, and the participants receiving the samples

Each distribution for aqueous-extractable VOCs, purgeable VOCs, SVOCs, or metals contains a maximum of four SPC-spiked samples and a matrix blank, each labeled by the SPC as described above. If four SPC-spiked samples are distributed, one is designated for use as the MS and MSD (additional aliquots of this sample may be provided), two are duplicates, and the remaining sample is unique. One or more of these samples may be eliminated by the RCRA PDP coordinator with approval from CBFO if it appears that the RCRA PDP goals can be achieved with fewer samples.

For VOCs and SVOCs, samples may be supplied as a preweighed aliquot in a separate container or as separate vials of waste matrix and spiking solutions. Instructions for preparation of analytical aliquots will be supplied. For VOCs, the aqueous-extractable analytes (alcohols, ketones, and pyridine) will be supplied separately from the balance of the VOCs (purgeable analytes). An identical complete backup set will be provided to allow re-analysis in the event of accidental destruction, loss of sample, or obvious laboratory error.

For metals samples, the target analytes will have been added to the matrix by the SPC, so participating laboratories may aliquot the samples according to the requirements of their internal procedures. Sufficient sample weight will be provided to permit withdrawal of multiple aliquots.

The RCRA PDP coordinator shall ensure that the SPC delivers the PDP samples to each of the laboratories participating in the PDP. The coordinator will inform all participants of developing PDP schedules and will give approximately two weeks notification of the PDP sample shipping date. The PDP samples will be sent to the attention of those individuals and addresses previously provided to the coordinator. Changes may be made to the addressee information by notifying the coordinator in writing at least 48 hours before the scheduled shipping date.

4.0 ANALYTICAL AND DATA REPORTING REQUIREMENTS

This section describes activities required of the participating laboratories with respect to PDP sample receipt, analysis, and reporting.

4.1 Sample Receipt and Chain-of-Custody

The following section describes procedures for sample receipt and management by participating laboratories (participants).

1. Immediately upon receipt of the samples, the participant shall locate the COC form. Appendix A contains an example COC form.
2. The participant verifies that the samples actually received match those listed on the COC form by both serial number and physical description. The participant also verifies that the samples have not been damaged during shipping.
 - a) If a discrepancy is noted, the participant shall notify the RCRA PDP coordinator immediately. The participant shall accept delivery by noting the discrepancies on the COC form and signing it, and shall await further instructions from the coordinator.
 - b) If no discrepancies are noted, the participant shall indicate receipt by signing the COC form. Completion of the COC form establishes the validated time of sample receipt (VTSR).
3. The participant shall return a *copy* of the COC form to the RCRA PDP coordinator within 24 hours of sample receipt and retain the *original* as the COC QA record for the samples. A copy of the COC form should also be forwarded to the SPC as stated in the instructions accompanying the samples.

4.2 Analysis

The participating laboratories analyze the contents of each sample according to the instructions provided with the samples, using the procedures that are used in the WIPP TRU waste characterization program. These procedures shall have been identified, documented, and approved within the facility's system for control of procedures implemented in compliance with the relevant requirements of the WAP and QAPD.

Preparation of the VOC and SVOC PDP samples requires adding an aliquot of a standard solution to a specified mass of matrix material. The site-approved procedures for analysis of VOCs and SVOCs may require a different sample mass than is specified in the analytical instructions provided with the PDP samples. If the mass of matrix material is varied, the solution volume shall be adjusted to maintain the original mass-to-concentration relationship.

To assess possible matrix effects or the effects of an interferent, aliquots of the designated MS/MSD samples are spiked with known quantities of participant-selected analytes. The MS and MSD samples should be spiked identically. The spiking levels for the MS and MSD analyses should be the corresponding PRQL of each of the spiked analytes. That is, the amount of spike should result in an increase in concentration of the target analyte equal to the PRQL. The minimum number of target analytes to spike for each category of analysis is as follows:

Category of analysis	Minimum number of spiked analytes
Aqueous-extractable VOCs	3
Purgeable VOCs	5
SVOCs	3
Metals	All

When analyzing the MS and MSD samples, the laboratory must analyze and report each target analyte without regard to whether it was used as one of the spiking analytes. In cases where TICs are identified in VOC or SVOC samples, the TIC determination does not need to be conducted in the MS/MSD analyses.

Any additional instructions that accompany the PDP samples shall also be followed (e.g., instructions for handling a noninterfering matrix).

Analyses should be completed and reported as soon as possible, but in any case, all required analytical data must be forwarded to the RCRA PDP coordinator within 28 calendar days after the VTSR, except as noted below.

If a participant's analyses will not be reported by the due date and the participant desires an extension, they must notify the RCRA PDP coordinator *in writing* (e-mail, fax, etc.) as soon as possible and request an extension. The coordinator will forward the request with a recommendation to the CBFO; the request will be either granted or rejected in writing by the CBFO. All extensions must be requested and granted before the due date. If an extension has not been granted prior to the due date, the coordinator may make the actual identity and concentrations of the analytes in the PDP samples known at any time thereafter. Once the known concentrations are released, any laboratory that has not yet reported may not be able to use these data to qualify for analysis of WIPP samples.

4.3 Reporting

Each PDP sample shall be analyzed for the analytes designated for that type of sample. Participating laboratories send to the RCRA PDP coordinator a summary of the analytical results for all analytes listed in tables 1, 2, and 3 for each sample analyzed. The concentrations of any detected analytes are to be reported, including TICs for the VOC and SVOC analyses. The following specifications apply to the summary report:

- Reports shall be forwarded directly to the RCRA PDP coordinator. Express mail or overnight delivery service is preferred, but in any case, all analytical reports to the coordinator shall be postmarked or shipped by an overnight delivery service no later than 28 calendar days after VTSR (except as noted in section 4.2).
- Analytical reports shall be submitted for each sample received and for laboratory blanks and MS/MSDs that are analyzed in association with the PDP samples.

The spreadsheet templates illustrated in Appendix B should be used to report the data to the RCRA PDP coordinator. Blank copies of these spreadsheet files may be distributed with the PDP samples or may be obtained from the coordinator. The following information shall be included in the spreadsheet for each determination:

- Identification of the reporting laboratory
- Identification of the PDP distribution cycle and analyte category (e.g., SVOCs, metals, etc.) for which the data are being reported
- Identity of the sample by the identification number from the COC form
- Any additional identification assigned to the sample by the laboratory
- Identification of the procedures (i.e., preparation and determination), including issue date and revision number, used for the analysis of each analyte. This identification is intended to be the facility's designation for the internally approved and documented procedure for performing the analysis in question with a cross reference to an appropriate SW-846 method number (U.S. Environmental Protection Agency [EPA] *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*)
- Identification of instrument or analytical system (i.e., GC/MS-III, ICP-1, CVAA-instrument k). **NOTE:** Each system's identification should be unique and traceable to a number permanently fixed to the system (e.g., a serial number or U.S. Government property identification number)
- Identity and concentration of each analyte identified
- Identity and MDL for each target analyte not identified in the sample
- Identification of any target analyte listed in tables 1 or 2 for which the laboratory intentionally did not analyze the PDP sample; for example, if pyridine was analyzed only in the aqueous-extractable VOC sample, pyridine should be listed on the SVOC report as "Not Analyzed as an SVOC"
- Date and time of analysis
- Any comments the laboratory feels are relevant to interpreting the data
- Definitions of any flag codes used on the report forms

After this information has been entered into the spreadsheet, the spreadsheet should be write-protected with a password known only to the participant. The results of each of the individual analyses must be reported. The report shall include a copy of the COC forms for the samples as

they existed at the time of reporting. The total number of pages in the report shall be indicated. Both hardcopy and electronic versions of the result spreadsheets shall be submitted to the RCRA PDP coordinator. The hardcopy shall be considered the official record.

Corrections to data are accepted if forwarded in writing before the scoring report is completed. Data may also be corrected by fax if followed by express mail or overnight courier transmission of the original hard copy. Verbal corrections to data will not be accepted. All corrections submitted shall include a clear explanation of the cause of the original error.

All compounds that exceed the PRDL or MDL must be qualitatively identified and an estimate of the concentration included. All compounds present at concentrations that exceed a calibration range or the PRQL must be quantified, even if multiple dilutions of the sample must be analyzed (see tables 1, 2, and 3 for PRDLs and MDLs). There is no requirement that concentrations of compounds and/or metals in the PDP samples be limited to any specific ratio range. (The RCRA PDP coordinator ensures that the ratios of analyte concentrations are not so large as to be likely to cause instrument contamination.)

Concentrations must be reported in milligrams per kilogram using sample reporting criteria specified in the WAP. The weight basis for all PDP samples is “as received” or “wet weight.”

The reports shall be reviewed and signed by a laboratory staff member assigned this responsibility. Reports should contain any other information that the laboratory feels is relevant to evaluation of the data.

4.4 Analytical Records

The requirement to submit only summary data for scoring does not relieve the laboratory of the requirement to maintain appropriate analytical records and documentation. The records generated during the analysis of the PDP samples are QA records. They must be maintained in a traceable and auditable condition. Storage conditions and duration must meet the requirements of the QAPD and other implementing QA documents and procedures.

5.0 EVALUATION OF PERFORMANCE DATA

Analytical performance is evaluated separately for the following analyte categories: purgeable VOCs, aqueous-extractable VOCs, SVOCs, and metals. Based on a point-scoring system, a participating laboratory will receive a “pass/fail” rating for each category for which results were submitted. The hardcopy results shall be considered the official record. Any discrepancies between the hardcopy and the electronic versions identified by the RCRA PDP coordinator may be brought to the participant's attention for resolution.

Scoring proceeds similarly for all analyte categories. As described in the following sections, scores are calculated for the following classes: performance on blanks, accuracy of quantitation, precision of quantitation of duplicates, and precision of replicate determinations. Matrix blanks are compared with PRQLs, single samples are scored for quantitation accuracy, duplicate samples are scored for quantitation accuracy and precision, and the MS/MSD samples (one plain and two spiked identically at the PRQLs for selected analytes) are scored for quantitation accuracy, duplicate precision (for spiked analytes), and replicate precision (for unspiked analytes). Scoring calculations become intricate and complex because in any given PDP sample, an analyte may be (1) absent, (2) present, but at a concentration less than the PRQL, or (3) present in excess of the PRQL. Scoring proceeds differently in each case and depends on whether the sample is a single, duplicate, or MS/MSD.

Scoring is conducted by awarding points for meeting the scoring criteria. In some instances, points may be deducted for failing to meet the criteria. Points awarded for all analytes within an analyte category must total 90 percent or more of the possible points, and points awarded to each analytical method must also total at least 90 percent of the points possible for that method in order for the category to pass the PDP cycle.

5.1 Performance on Blanks

Purpose: Analytical results for blanks are used to determine the presence of contamination problems and to quantify those problems if any exist. Analytical data from the analysis of all blanks must be reported. The data from laboratory blanks may be used in interpreting the significance of anomalous or incorrect data reported for the other PDP samples.

Criterion: None of the target analytes should be present in the blank analyses at levels exceeding 50 percent of the PRQL. Ideally, blank concentrations should be less than the MDL or PRDL.

Evaluation Method: Acceptable blank performance is based on the data for all detected target analytes and their concentrations relative to their PRQLs listed in tables 1, 2, and 3. Interpretation of reported values depends on the known (certified) concentration of an analyte in the blank (C_{known}). The scoring parameter, F_{PRQL} , is calculated as follows:

$$F_{PRQL} = \frac{C_{meas} - C_{known}}{PRQL} \times 100 \quad (1)$$

where:

F_{PRQL} = fraction of the PRQL represented by the measured concentration, expressed as a percentage, for an analyte of interest in the blank.

C_{meas} = measured concentration or detection limit of the analyte of interest in the blank (mg/kg).

$PRQL$ = program required quantitation limit for the analyte of interest (mg/kg).

C_{known} = certified value of compound in the PDP sample (mg/kg).

Scoring: Possible points are –1 to +1 per analyte.

- If $F_{PRQL} < 50\%$, award one point.
- If $F_{PRQL} \geq 50\%$, deduct one point.

Actions: If a preponderance of evidence from the participating laboratories indicates that a specific analyte is present in the blank at levels exceeding 50 percent of the PRQL, the blank will be considered contaminated. The analyte data will be deemed unusable and the analyte will be deleted from consideration in the current cycle of the PDP.

5.2 Detection and Accuracy

Purpose: Analytical results for PDP samples of known concentration are used to determine the accuracy with which a laboratory can quantitate the target analytes.

Criterion: Reported results for the target analytes present at concentrations greater than the PRQL should not deviate from the certified values by more than the accuracy ranges given in tables 1, 2, and 3.

Evaluation Method: The recovery for each of the target analytes is calculated relative to the known value. For individual PDP samples, the certified value is the known value, whereas for MS/MSD samples, the amount of spike added is the known value. For individual PDP samples (and unspiked analytes in MS/MSD samples):

$$\%R = \frac{C_{meas}}{C_{known}} \times 100 \quad (2)$$

where:

$\%R$ = percent recovery of the analyte of interest from the PDP sample.

C_{meas} = measured concentration of the analyte of interest from the laboratory's analysis of the PDP sample (mg/kg).

C_{known} = certified value of the analyte of interest in the PDP sample (mg/kg).

For spiked analytes in MS/MSD samples:

$$\%R = \frac{CS_{meas} - C_{meas}}{CS_{known}} \times 100 \quad (3)$$

where:

- $\%R$ = percent recovery of the analyte of interest from the PDP sample.
- CS_{meas} = measured concentration of the analyte of interest from the laboratory's analysis of the matrix-spiked sample (mg/kg).
- C_{meas} = measured concentration of the analyte of interest from the laboratory's analysis of the corresponding unspiked PDP sample (mg/kg).
- CS_{known} = concentration of the analyte of interest attributable to the addition of the matrix spike (mg/kg), approximately equal to the PRQL (see section 4.2).

Scoring for individual samples: Possible points are 0 to 3 per analyte if the known concentration is greater than or equal to the PRQL, or 0 to 1 if the certified concentration is between the PRQL and 10 percent of the PRQL. If the certified concentration is less than 10 percent of the PRQL, one point may be deducted for false detection.

- If C_{known} is $\geq PRQL$, then
 - If C_{meas} is $>$ the PRDL or MDL, award two points for detection.
 - If $\%R$ is within the accuracy range given in tables 1, 2, and 3, award one point for accuracy.
- If C_{known} is $< PRQL$ and C_{known} is ≥ 10 percent of $PRQL$, then
 - If C_{meas} is $>$ the PRDL or MDL (i.e., reported as a positive detection), award one point for detection.
- If C_{known} is < 10 percent of $PRQL$, then
 - If C_{meas} is $\geq PRQL$, deduct one point for false positive detection.

Scoring for spiked analytes in MS/MSD samples: Possible points are -2 to $+3$ per analysis.

- If CS_{meas} is $>$ the PRDL or MDL, award two points for detection.
- If CS_{meas} is $<$ the PRDL or MDL, deduct two points for non-detection.
- If $\%R$ is within the accuracy range given in tables 1, 2, and 3, award one point for accuracy.
- If $\%R$ does not meet the accuracy range given in tables 1, 2, and 3, deduct one point for accuracy.

Actions: If all participating laboratories report that a specific analyte falls outside the accuracy range for $\%R$ in the same direction, then those data may be judged as inappropriate for inclusion in the current cycle of the PDP.

5.3 Precision of Duplicates

Purpose: Analytical results for blind duplicate PDP samples of known concentration are used to determine the precision with which a laboratory can quantitate the target analytes.

Criterion: For the target analytes present at concentrations greater than the PRQL, the difference between determinations from blind duplicate samples should not exceed the values given in tables 1, 2, and 3 for precision.

Evaluation Method: If the target analyte is known to be present at a concentration greater than the PRQL, the relative percent difference between duplicate determinations is calculated as follows:

$$RPD = \frac{|C1_{meas} - C2_{meas}|}{\left(\frac{C1_{meas} + C2_{meas}}{2}\right)} \times 100 \quad (4)$$

where:

RPD = relative percent difference between the measured values from two duplicate samples.

$C1_{meas}$ = measured concentration of the analyte of interest in duplicate sample 1 (mg/kg).

$C2_{meas}$ = measured concentration of the analyte of interest in duplicate sample 2 (mg/kg).

Scoring for plain PDP samples: Possible points are 0 to 1 per pair of analyses.

- If RPD meets the precision standard in tables 1, 2, and 3, award one point for precision.

Scoring for MS/MSD samples (spiked analytes): Possible points are -1 to +1 per pair of analyses.

- If RPD meets the precision standard in tables 1, 2, and 3, award one point for precision.
- If RPD does not meet the precision standard in tables 1, 2, and 3, deduct one point for precision.

Actions: None.

5.4 Precision of Replicates

Purpose: Analytical results for replicate (i.e., triplicate or more) analyses of PDP samples of known concentration will be used to determine the precision with which a laboratory can

quantitate the target analytes. Triplicate analyses occur for nonspiked analytes in the MS/MSD sample set.

Criterion: The sample standard deviation of replicate analyses should not exceed the values given in tables 1, 2, and 3 for precision.

Evaluation Method: Replicate determinations for each sample are used to calculate the relative percent standard deviation for each of the target analytes as follows:

$$\%RSD = \frac{s}{C_{avg}} \times 100 \quad (5)$$

where:

$\%RSD$ = relative standard deviation of the replicate determinations, expressed as a percentage of the average concentration.

s = sample standard deviation of the replicate determinations.

C_{avg} = average concentration of the analyte of interest in replicate determinations (mg/kg).

Scoring: Possible points are 0 to 1 per set of replicate analyses.

- If $\%RSD$ meets the precision standard in tables 1, 2, and 3, award one point for precision.

Actions: None.

5.5 Tentatively Identified Compounds (TICs)

Purpose: The possibility that other analytes may be present in a VOC or SVOC PDP sample in addition to those on the target analyte list (tables 1, 2, and 3) requires that a participating laboratory report all detected analytes, not just the target analytes.

Criterion: For VOC and SVOC, additional nontarget analytes shall be correctly identified.

Evaluation: Nontarget analytes need only be detected, i.e., the reported value must be greater than the instrument or method detection limit.

Scoring: Possible points are -0.1 or +0.1 for each TIC, up to 10 TICs.

- If a TIC is known to be present, then
 - If it is detected, award 0.1 points.
 - If it is not detected or misidentified, deduct 0.1 points.

Actions: None.

5.6 Overall Performance

Purpose: The WAP requires that laboratories characterizing waste for shipment to WIPP demonstrate their ability to identify and quantitate analytes in tables 1, 2, and 3. Performance on the PDP samples reveals problems that may affect a laboratory's ability to analyze the target analytes.

Criterion: In a single PDP cycle, a laboratory must score at least 90 percent of the points possible for a given analyte category in order to pass that category in the PDP cycle.

Evaluation Methods: Analyses of the PDP samples are scored using as many scoring classes as possible. The score for an individual analyte may be a composite of several scoring classes, depending on the type of PDP sample.

Scoring for blanks:

- If F_{PRQL} is <50 percent of the PRQL, possible points are +1 per analyte (for detection).
- If F_{PRQL} is \geq 50 percent of the PRQL, possible points are -1 per analyte (for false positive detection).

Scoring for single samples:

- If C_{known} is $\geq PRQL$, possible points are 0 to 3 per analyte (0 to 2 for detection, 0 to 1 for accuracy).
- If C_{known} is $< PRQL$ and C_{known} is \geq 10 percent of $PRQL$, possible points are 0 to 1 per analyte (for detection).
- If C_{known} is <10 percent of $PRQL$, then if C_{meas} is $> PRQL$, possible points are -1 to 0 per analyte (for false positive detection).

Scoring for duplicate samples (two analyses):

- If C_{known} is $\geq PRQL$, possible points are 0 to 7 per analyte (0 to 4 for detection, 0 to 2 for accuracy, 0 to 1 for precision).
- If C_{known} is $< PRQL$ and C_{known} is \geq 10 percent of $PRQL$, possible points are 0 to 2 per analyte (for detection only).
- If C_{known} is <10 percent of $PRQL$, then if C_{meas} is $> PRQL$, possible points are -2 to 0 per analyte (for false positive detection).

Scoring for MS/MSD samples (three analyses):

- If the analyte has been spiked (one unspiked analysis, two spiked analyses), then
 - If C_{known} is $\geq PRQL$, possible points are -7 to 10 per analyte (-4 to 6 for detection, -2 to 3 for accuracy, -1 to 1 for precision).

- If C_{known} is $< PRQL$ and C_{known} is ≥ 10 percent of $PRQL$, possible points are -7 to 8 per analyte (-4 to 5 for detection, -2 to 2 for accuracy, -1 to 1 for precision).
- If C_{known} is < 10 percent of $PRQL$, then if C_{meas} is $> PRQL$ for the unspiked analysis, possible points are -8 to $+7$ per analyte (-1 to 0 for false positive detection, -4 to 4 for detection, -2 to 2 for accuracy, -1 to 1 for precision).
- If the analyte has not been spiked (three unspiked analyses), then
 - If C_{known} is $\geq PRQL$, possible points are 0 to 10 per analyte (0 to 6 for detection, 0 to 3 for accuracy, 0 to 1 for precision).
 - If C_{known} is $< PRQL$ and C_{known} is ≥ 10 percent of $PRQL$, possible points are 0 to 3 per analyte (for detection only).
 - If C_{known} is < 10 percent of $PRQL$, then if C_{meas} is $> PRQL$, possible points are -3 to 0 per analyte (for false positive detection).

Example Point Distribution: A laboratory analyzes five samples for seven target analytes (e.g., aqueous-extractable VOCs). There are no TICs. Known concentrations are considered “normal” if greater than the PRQL, “low” if less than the PRQL but greater than 10 percent of the PRQL, and “absent” if less than 10 percent of the PRQL. The characteristics of the samples are as follows:

- Sample 1 is a blank. All analytes are absent.
- Sample 2 is a single sample. Two analytes have low concentrations. The remaining analytes have normal concentrations.
- Samples 3 and 4 are blind duplicates. Two analytes have low concentrations, one analyte is absent, and the remaining analytes have normal concentrations.
- Sample 5 is used for MS/MSD analyses. Three analytes are spiked. Two analytes (including one of the spiked analytes) have low concentrations, and the remaining analytes have normal concentrations.

The laboratory can score a maximum of 117 points, broken down as indicated by subscripts:

- Sample 1 (blank): $(7 \times 1)_{\text{absent}} = 7$ points
- Sample 2 (single): $(2 \times 1)_{\text{low}} + (5 \times 3)_{\text{normal}} = 17$ points
- Samples 3 and 4 (duplicates): $(2 \times 2)_{\text{low}} + (2 \times 0)_{\text{absent}} + (4 \times 7)_{\text{normal}} = 32$ points
- Sample 5 (MS/MSD): $(2 \times 10)_{\text{spiked,normal}} + (1 \times 8)_{\text{spiked,low}} + (1 \times 3)_{\text{plain,low}} + (3 \times 10)_{\text{plain,normal}} = 61$ points

The laboratory’s actual score is determined using the scoring criteria in sections 5.1 – 5.5. In this example, a passing score is 117×90 percent = 106 points.

Sample or Analyte Disqualification: If the preponderance of evidence from the participating laboratories indicates that the concentration of a specific analyte in a sample has not been

certified accurately enough to demonstrate compliance with the criteria of the PDP, the RCRA PDP coordinator may exclude that analyte from that cycle of the PDP. This decision will be based on a comparison of the values reported by the participants to the reference value for the analyte provided by the SPC. In making this determination, the coordinator will consider whether the participant(s) has previously demonstrated the capability to correctly quantitate the analyte in question in a similar sample matrix by the same method(s) and whether sufficient data are available for an adequate statistical test.

Actions: The action level is a score of less than 90 percent of the possible points in an analyte category or for an analytical method. If a laboratory exceeds the action level (scores less than 90 percent), corrective actions will be necessary. The site project manager shall be responsible for ensuring that appropriate corrective actions are implemented. Any laboratory that has exceeded an action level shall discontinue the use of any potentially affected characterization data for certification of WIPP wastes. The laboratory may not use such potentially affected characterization data for certification of WIPP wastes until it obtains CBFO's approval to do so. To obtain this approval, the laboratory must submit a report to CBFO containing the following items:

1. Results of an investigation of the cause of the failure(s).
2. Description of any corrective actions completed and/or proposed as a result of the investigation.
3. Supporting data sufficient to demonstrate that the same problems will not recur.
4. A plan and schedule for the disposition for all potentially affected waste characterization data, for example, any data collected prior to the first PDP cycle, between a successful and a failed PDP cycle, or between completion of a PDP cycle and the issuance of the report for that cycle. (Such data shall be treated as potentially nonconforming under the facility's QA program.)
5. An assessment of the impact of the participating laboratory's "Not Approved" status for RCRA on waste characterization activities at the site.
6. A proposed mechanism for obtaining approved status from CBFO, including a request for approval in a supplemental PDP cycle or for approval with waiver of a supplemental cycle.

CBFO may elect to grant conditional approval for a laboratory to perform waste characterization analyses for this program if such conditional approval will not compromise the overall quality of the data being generated for the program. Such conditional approval may be granted if:

- The laboratory's failure to meet criteria was limited to a very few compounds (possibly even a single compound).
- CBFO has reason to believe that the error is systematic and likely to be correctable after appropriate corrective actions.

- Limitations and conditions can be placed on the approval to guarantee that suspect data will not be used in the program.

The potential causes for exceeding an action level are many and varied. Rather than cease operations, the laboratory may choose to proceed at risk until corrective actions have been completed. The laboratory should recognize that data obtained before the action level was exceeded and any data obtained at risk may be found to be unacceptable to WIPP.

6.0 REPORTING OF PERFORMANCE DATA

6.1 Summary of Data

The RCRA PDP coordinator shall review and evaluate the results from all participants, summarize them in a scoring report, and deliver this report to DOE-CBFO within approximately five weeks after receipt of the last laboratory data set. The report summary shall include the values reported by the laboratories, the certified target-analyte values, the acceptable ranges for accuracy and precision for each target analyte, and the approval status of each individual laboratory.

6.2 Distribution of Reports

Copies of the summary report shall be distributed to the DOE Operations Offices involved, to the participating laboratories, and to other individuals and organizations that CBFO deems appropriate. The CBFO shall also provide separate written notification to the DOE Operations Offices regarding the performance and approval status of their participating laboratories.

6.3 Laboratory Status

The CBFO, in conjunction with the RCRA PDP coordinator, evaluates laboratory performance and approves laboratories for participation in the WIPP waste characterization program. Approval status is determined for each analyte category (i.e., metals, aqueous-extractable VOCs, purgeable VOCs, and SVOCs) and laboratory analytical method. If a quality-affecting change is made to the analytical method, the method is considered new and must be qualified in a PDP cycle before it can be used to perform WIPP analyses.

In accordance with the WAP, once the CBFO has determined that a laboratory and method are approved, that status shall remain in effect for 13 months (i.e., 12 months plus a one-month grace period). All laboratories must participate in the annual primary cycle in order to remain qualified to perform WIPP analyses. A timely response in the annual primary cycle will ensure that a laboratory will not exceed its qualification time limit.

The qualification period for a laboratory begins with the date that analyses in a PDP cycle are completed. After the end of the 13th month, a laboratory that has not yet successfully completed their analyses of PDP samples to requalify their method(s) may choose to proceed at risk with WIPP analyses or cease operations. The laboratory should recognize that data obtained at risk may be found to be unacceptable to WIPP. Data generated at risk cannot be used for certifying waste for shipment to WIPP until:

- The system used to collect the data passes the PDP, and
- The data have been reconciled through the dispositioning of a nonconformance report.

6.4 Quality Assurance Records

The minimum QA records for the RCRA PDP are identified and listed below in accordance with the QAPD requirements. In addition, the RCRA PDP coordinator may determine that records of other program activities are QA records and enter them into the QA records system with the same level of control and maintenance.

These QA records may be organized by RCRA PDP Plan revision, by PDP cycle, or other principle, as applicable. These records are nonpermanent records and shall be maintained in accordance with the QAPD requirements. Records disposition, when applicable, will be in accordance with CBFO requirements and approved procedures. All QA records identified in this plan shall be stored in accordance with record storage requirements in the QAPD. Access to QA records is limited to personnel involved in the program or having related QA or records custodial responsibilities.

QA records for the RCRA PDP include the following:

- Procedures (for example, management procedures, operating procedures, or work instructions) (all revisions)
- PDP plans (all revisions)
- Procurement records
- Personnel qualification records required by section 2.4 of this plan
- Records of set-up for each cycle (planned target analytes and concentrations, notification letters, final certified values with verification data, shipping records, and other correspondence)
- Participant analytical reports and supporting forms for each cycle (analytical data report forms, COC records, transmittal letters)
- Scoring reports for each cycle
- Reviews of corrective actions and supporting data and recommendations made to CBFO for each cycle
- Software documentation for programs written for the RCRA PDP, as defined in the applicable approved software procedure (each version, each program)

7.0 GLOSSARY

ACCURACY – The degree of agreement between a measured value and an accepted reference or the true value. Accuracy is determined as the percent recovery.

ACTION LEVEL – A numerical criterion that must be met for a type of analysis, e.g., a fraction of %R values that must fall within the respective QAOs. Failure to meet this criterion may result in a conclusion that the laboratory is unable to adequately perform a specific type of analysis.

ANALYSIS DATE/TIME – The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE – The element, ion, or compound an analysis seeks to determine; the element of interest.

ANALYTICAL METHOD – The sample preparation and instrumentation procedures or steps that must be performed to estimate the quantity of analyte in a sample.

BLIND AUDIT SAMPLE – A sample of known composition provided as a single-blind sample to the analytical laboratory, which DOE uses to evaluate analytical laboratory performance. Blind audit samples are distributed to participating laboratories as part of the PDP.

CHAIN-OF-CUSTODY – A set of procedures established to ensure that the integrity of the sample and that of the sample data are maintained.

CORRECTIVE ACTION – Measures taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.

CYCLE – An annual process that implements the RCRA PDP. The cycles are numbered using both a numeric and alpha character. The numeric designation stands for all cycles initiated within the same year. The alpha designator indicates the primary cycle and each supplemental cycle. For example, Cycle 8A is the primary cycle of the year, with Cycle 8B, Cycle 8C, etc., being supplemental cycles administered during the same year.

DUPLICATE – A second aliquot of a sample that is treated the same as the original sample to determine the precision of the method.

INSTRUMENT DETECTION LIMIT – The minimum signal that an instrument can detect with 99 percent confidence that the analyte concentration is greater than zero.

INTERFERENTS – Substances that affect the analysis for the element or compound of interest.

LABORATORY BLANK – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample analysis. The laboratory blank is used to assess contamination resulting from the laboratory sample preparation and analytical process.

LABORATORY CONTROL SAMPLE – A control sample of known composition. Laboratory control samples are analyzed using the same analytical methods employed for the program samples received.

MATRIX SPIKE – An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

MATRIX SPIKE DUPLICATE – Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. Matrix spike duplicates are used to document the precision and bias of a method in a given sample matrix.

METHOD DETECTION LIMIT – The minimum concentration of an analyte that can be measured and reported for a given method with 99 percent confidence that the analyte concentration is greater than zero. MDL is determined from analysis of a sample in a given matrix type containing the analyte of interest. The maximum values for MDLs permissible for the program are presented in tables 1 and 2.

PARTICIPANT – A measurement facility seeking to certify its RCRA analytical processes under the RCRA PDP. A measurement facility requests permission to be a participant in accordance with the procedure found in the RCRA PDP Plan.

PDP MANAGER – An individual responsible for overall performance of, and coordination among, the three PDPs (HSG, RCRA, and NDA).

PDP SAMPLE – A blind audit sample prepared specifically for use in the PDP.

PRECISION – A measure of mutual agreement among individual measurements of the same property made under prescribed similar conditions; often expressed as a standard deviation or relative percent difference.

PROCEDURE – A detailed, step-by-step description of the sequence of actions to be followed to perform a given task or activity. The term procedure also includes instructions and drawings. If followed in sequence, a procedure provides enough information that a trained person could complete the covered task without additional information.

PROGRAM COORDINATOR – A CBFO-designated organization that administers and coordinates PDP functions.

PROGRAM REQUIRED DETECTION LIMIT – The maximum values for instrument detection limits permissible for metals analyses. PRDLs are presented in table 3.

PROGRAM REQUIRED QUANTITATION LIMIT – Level of analyte quantitation under the WAP that causes specified actions. The PRQLs establish the values on measured analyte concentrations that meet program goals or cause specific actions to be taken. For example, for each target analyte in a waste stream, the PRQL is compared with the UCL₉₀. If the UCL₉₀ for

the analyte is greater than or equal to the PRQL, the EPA hazardous waste number for that analyte may be assigned to the waste stream. PRQLs are presented in tables 1, 2, and 3.

QUALITY ASSURANCE – Planned and systematic actions necessary to provide adequate confidence that an item will perform satisfactorily in service.

QUALITY ASSURANCE OBJECTIVES – The characteristics of data that are associated with their ability to satisfy a given purpose or objective. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability.

RCRA PDP COORDINATOR – An individual assigned by the PDP manager who administers and coordinates PDP functions, such as PDP sample component preparation, subcontractor oversight, scheduling, scoring, and report summary generation.

RECOVERY – The numerical ratio of the amount of analyte measured by the laboratory method divided by the known amount of analyte added to or known to be present in the matrix to be analyzed, expressed as a percent.

SAMPLE – A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE PREPARATION CONTRACTOR – An independent contractor responsible for the actual preparation and shipping of the PDP samples.

TARGET ANALYTES – Those VOCs, SVOCs, metals, and nonmetallic elements identified by the program as analytes. Target analytes for the program are listed in tables 1–3.

TENTATIVELY IDENTIFIED COMPOUNDS – Nontarget analytes identified using gas chromatography/mass spectrometry (GC/MS). These reported concentrations will have a higher uncertainty associated with them than the reported target analyte concentrations.

TRANSURANIC WASTES – Laboratory and process wastes that contain alpha-emitting radionuclides of atomic number greater than 92 (e.g., the isotopes of plutonium), have half-lives longer than 20 years, and are present in concentrations greater than 100 nanocuries per gram of waste.

VALIDATED TIME OF SAMPLE RECEIPT – The date on which a sample is received at the analytical facility, as recorded on the COC form.

VOLATILE ORGANIC COMPOUNDS – For the purposes of the program, those VOCs listed in table 1 and any additional compounds tentatively identified by the VOC analytical procedures used to satisfy program requirements.

8.0 REFERENCES

- DOE/CBFO-94-1012, *Quality Assurance Program Document*. Current Revision. U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico
- DOE/CBFO 95-1076, *Performance Demonstration Program Plan for the Analysis of Simulated Headspace Gases*. Current Revision. U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico
- DOE/CBFO-01-3107, *Performance Demonstration Program Management Plan*. Current Revision. U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico
- DOE/CBFO-01-1005, *Performance Demonstration Program Plan for Nondestructive Assay of Drummed Wastes for the TRU Waste Characterization Program*. Current Revision. U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico
- DOE/CBFO-01-1006, *Performance Demonstration Program Plan for Nondestructive Assay of Boxed Wastes for the TRU Waste Characterization Program*. Current Revision. U.S. Department of Energy, Carlsbad Field Office, Carlsbad, New Mexico
- NM4890139088-TSDF, *Waste Isolation Pilot Plant Hazardous Waste Facility Permit*. NMED 1999, as modified. New Mexico Environment Department, Santa Fe, New Mexico
- SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. Current Revision. U.S. Environmental Protection Agency

APPENDIX A

Example Chain-of-Custody Form

Sample Identification and Chain of Custody Form - WIPP PDP Cycle XX

Ship To: Participant XYZ Shipping Address	Ship From: Sample Preparation Contractor Address Phone Contact: Contact Name
Attention: Identified Recipient	

Sample Identification	Metals					Date Sampled	Analysis Required
ME 9621	1					MM/DD/YYYY	See instruction sheets
ME 9621 B	1					MM/DD/YYYY	See instruction sheets
ME 7010 *	1					MM/DD/YYYY	See instruction sheets
ME 7010 B *	1					MM/DD/YYYY	See instruction sheets
ME 6005	1					MM/DD/YYYY	See instruction sheets
ME 6005 B	1					MM/DD/YYYY	See instruction sheets
ME 7513	1					MM/DD/YYYY	See instruction sheets
ME 7513 B	1					MM/DD/YYYY	See instruction sheets
ME 1009	1					MM/DD/YYYY	See instruction sheets
ME 1009 B	1					MM/DD/YYYY	See instruction sheets

Relinquished By	Date	Time	Received By	Date	Time	Condition of Contents
_____	_____	_____	_____	_____	_____	_____
Relinquished By	Date	Time	Received By	Date	Time	_____
_____	_____	_____	_____	_____	_____	_____
Relinquished By	Date	Time	Received By	Date	Time	_____
_____	_____	_____	_____	_____	_____	_____

**Comments: Utilize ME 7010 and ME 7010 B for MS & MSD.*

APPENDIX B

Example Data Reporting Forms

Performance Demonstration Program for the Analysis of RCRA Constituents Cycle XX
Simulated Solidified Solid Waste Analysis
PURGEABLE VOCS (Sample 3)

Laboratory Name:		Report Page:	3
PDP Sample ID:		Total Pages in Report:	8
Lab Sample ID:		PDP Distribution (mm/dd/yyyy):	
Analytical Instrument ID:			

Analyte	Result (mg/kg)	Flag ¹	D.L. ⁵ (mg/kg)	Method Identification		Analysis		Comment
				Site #	SW-846 #	Date	Time	
Acetone ³								
Benzene								
Bromoform								
Carbon disulfide								
Carbon tetrachloride								
Chlorobenzene								
Chloroform								
1,4-Dichlorobenzene ²								
1,2-Dichlorobenzene ²								
1,2-Dichloroethane								
1,1-Dichloroethene								
trans-1,2-Dichloroethylene								
Ethyl benzene								
Ethyl ether ³								
Methyl ethyl ketone ³								
Methylene chloride								
1,1,2,2-Tetrachloroethane								
Tetrachloroethylene								
Toluene								
1,1,1-Trichloroethane								
1,1,2-Trichloroethane								
Trichloroethylene								
Trichlorofluoromethane								
1,1,2-Trichloro-1,2,2-trifluoroethane								
Vinyl chloride								
m- & p-Xylene								
o-Xylene								
TICs ⁴								

Notes:

- Flags are defined in the WAP.
- If analyzed as an SVOC, enter "n/a" in the Result column and make a note in the Comment column.
- If analyzed as an AVOC, enter "n/a" in the Result column and make a note in the Comment column.
- Insert rows as needed to accommodate all TICs detected.
- D.L. = detection limit.

Additional Comments:

Approval: _____
Signature Title Date

Performance Demonstration Program for the Analysis of RCRA Constituents Cycle XX
Simulated Solidified Solid Waste Analysis
METALS (Sample 1)

Laboratory Name:		Report Page:	1
PDP Sample ID:		Total Pages in Report:	8
Lab Sample ID:		PDP Distribution (mm/dd/yyyy):	
Analytical Instrument ID:			

Analyte	Result (mg/kg) ³	Flag ¹	D.L. ² (mg/kg)	Method Identification		Analysis		Comments
				Site #	SW-846 #	Date	Time	
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Chromium								
Lead								
Mercury								
Nickel								
Selenium								
Silver								
Thallium								
Vanadium								
Zinc								

Comments:

Notes:

1. Flags are defined in the WAP.
2. D.L. = detection limit.
3. Results should be expressed as wet weight.

Approval: _____
Signature

Title

Date

Performance Demonstration Program for the Analysis of RCRA Constituents Cycle XX
Simulated Solidified Solid Waste Analysis
SVOCs (Sample 1)

Laboratory Name:		Report Page:	1
PDP Sample ID:		Total Pages in Report:	8
Lab Sample ID:		PDP Distribution (mm/dd/yyyy):	
Analytical Instrument ID:			

Analyte	Result (mg/kg)	Flag ¹	D.L. ⁵ (mg/kg)	Method Identification		Analysis		Comments
				Site #	SW-846 #	Date	Time	
m&p-cresol								
o-cresol								
1,4-Dichlorobenzene ²								
1,2-Dichlorobenzene ²								
2,4-Dinitrophenol								
2,4-Dinitrotoluene								
Hexachlorobenzene								
Hexachloroethane								
Nitrobenzene								
Pentachlorophenol								
Pyridine ³	N/A							
TICs ⁴								

Notes:

- Flags are defined in the WAP.
- If analyzed as a PVOC, enter "n/a" in the Result column and make a note in the Comment column.
- If analyzed as an AVOC, enter "n/a" in the Result column and make a note in the Comment column.
- Insert rows as needed to accommodate all TICs detected.
- D.L. = detection limit.

Additional Comments:

Approval: _____
Signature Title Date